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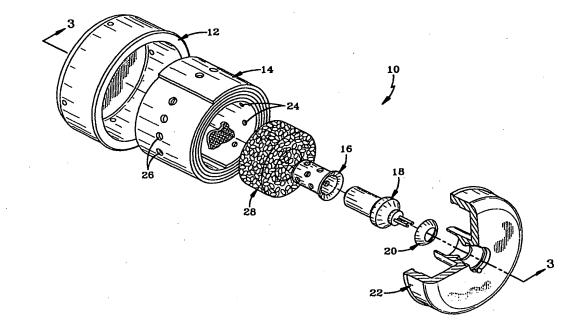
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(54) Title: INFLATOR FOR USE WITH GAS GENERANT COMPOSITIONS CONTAINING GUANIDINES



#### (57) Abstract

An inflator (10) for a vehicle airbag employing non-azide gas generants. A new filter (14) for airbag inflators is a metal ribbon coiled within the inflator house (12, 22). The metal ribbon comprises at least one segment of apertures and a segment of expanded metal.

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# INFLATOR FOR USE WITH GAS GENERANT COMPOSITIONS CONTAINING GUANIDINES

The present invention relates to an inflator used for inflating an airbag of a vehicle occupant restraint system, and more specifically for an inflator housing for use with gas generant compositions containing guanadines.

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The use of such protective gas-inflated airbags to cushion vehicle occupants in crash situations is now widely known and well documented and the requirements of a gas generant composition used in a vehicle airbag inflator are very demanding. Much effort has been expended on the development of inflator housings. The housings are the metal shells in which the gas generant compositions are placed and ignited when an airbag needs to be inflated. This housing is fitted with the airbag and together they form a critical part of any vehicle airbag system. Engineers of vehicle airbag systems understand that the design of the inflator housing can influence the performance of the gas generant and the overall protection of the vehicle occupant.

The gas generant composition must efficiently produce a relatively cool, non-toxic, non-corrosive gas which is easily filtered to remove solid and liquid combustion by-products. This filtering is needed to preclude damage to the inflatable airbag or injury to the occupant of the automobile. These requirements limit the applicability of many otherwise suitable chemical compositions, shapes and configurations from being used in automotive airbag inflators. Gas generants can also be used for fire

extinguishing. Recently, a number of companies have begun using the gases produced by solid energetic or pyrotechnique materials for fire extinguishing.

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The filter system of the present invention is a metal strip or ribbon with two regions having a plurality of apertures and a region of expanded metal. This metal strip is coiled and placed within the inflator housing between the gas generant and the gas exit portals of the housing. While the placement of a filter within the housing is conventional, the presently disclosed filter system provides unexpectedly improved results at minimal labor and material cost. The following is a brief discussion of conventional approaches to filtering combustion gases.

US 3 785 674 teaches an inflator housing that provides for the combustion gases to pass through a separating screen and then a steel wool filter and into a secondary reacting chamber which "reacts out" the undesirable combustion products while permitting the free nitrogen to pass therethrough.

US 3 958 949 teaches an inflator housing with a combustion chamber laterally surrounded by a cooling chamber. The solid coolant material contained in the cooling chamber consists of potassium perchlorate containing copper chromite as a decomposition catalyst. To prevent solid particles from being expelled from the generator, retaining grids are placed adjacent to the discharge apertures.

US 4 316 874 teaches a gas generator utilizing granular silicon carbide as a coolant. The generally cylindrical housing assembly has disposed within it two (2) cylindrically shaped stainless steel wire screens so that the interior of the housing is

partitioned into a cylindrical central space and a radially outer annular space. The annular space defined between the two wire screens is filled with granular silicon carbide.

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US 4 561 675 discloses an inflator that uses multiple filters and a tortuous gas path to remove particulates and cool the exhaust gas. More specifically, this patent teaches that the gas generant pellets be surrounded by an annular inner screen pack or combustion filter. The inflator also possesses an outer screen pack or filter that desirably includes a coarse layer adjacent the inner surface of the cylindrical housing.

In yet another air bag inflator design,

US 4 858 951 teaches an airbag inflator wherein the
gas generated by the combustion of the generant flows
radially through openings in a rigid cylindrical metal
tube which surrounds the gas generant pellets. The
gas then flows through a filter that is preferably

made of a plurality of layers of wire mesh, steel wool
and fiberglass.

US 5 016 914 teaches a hybrid inflator that uses a gas generant to heat an inert gas stored in a container within the inflator. The separation of solid residue from the generated gas is accomplished in part by a 180° turn at the end of the outer housing. At least some of the relatively large particles are inertially removed from the nitrogen gas stream and impinge and fuse against the doomed surface which is preferably coated with a high temperature grease which helps capture the particles.

Typically, the prior art filters have been made of wire gauze and/or inorganic fiber. The winding of

stainless steel screens, fine-mesh woven metal wire, close-woven metal wire, etc. in layers to form filters that function to cool the combustion gas and remove the combustion residue or slag is also known.

## BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is an exploded view of an inflator used in
the tests described herein and employing the inventive
filter system;

FIG. 2A is a top plan view of one embodiment of the metallic ribbon used to prepare the filter coil according to the invention;

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FIG. 2B is a top plan view of a second embodiment of the metallic ribbon; and

FIG. 3 is a side view in cross section of the inflator taken along line 3-3 of FIG. 1.

#### DETAILED DESCRIPTION OF THE INVENTION

The gas generant formulations used in the 5 inflator of this invention are formulated from the guanidine family of fuels such as guanidine nitrate (GN), triaminoquanidine nitrate (TAGN) and the like. The fuel component will typically comprise between about 45 and about 70 weight %, more preferably 10 between 50 and 60 weight %, of the gas generant composition, while the oxidizer system will typically comprise between about 35 and about 50 weight %, more preferably between 40 and 50 weight %, of the gas generant composition. Processing aids, such as 15 silicon dioxide, may also be used in formulating the gas generant pellets. Those skilled in the art understand that depending upon the particular oxidizers and fuels utilized, certain processing aids have beneficial properties over others.

The fuel useful in the gas generant used in the inflator of the present invention is a mixture of at least two guanidine fuels selected from guanidine nitrate (GN), nitroguanidine (NG), triaminoguanidine nitrate (TAGN), diaminoguanidine nitrate (DAGN) and monoguanidine nitrate (MGN). Guanidine or iminourea (CH5N3) has the structural formula:

$$H_2N-C < NH$$

Guanidine is soluble in water and alcohol, volatile and strongly alkaline. It forms many salts, e.g., nitrate and the like. Nitroguanidine is a white crystalline powder that is usually manufactured from

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calcium carbide via calcium cyanamide, dicyandiamide and guanidine nitrate which is converted to nitroguanidine by action of concentrated sulfuric acid. Nitroguanidine has the structural formula:

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$$H_2N-C \stackrel{NH}{\sim} NH^{\bullet}NO_2$$

Oxidizers useful in the gas generant compositions used in the inflator of the present invention include ammonium perchlorate and the alkali metal and alkaline earth metal nitrates such as strontium nitrate and sodium nitrate. The preferred oxidizer system is a mixture of strontium nitrate, sodium nitrate and ammonium perchlorate. Ammonium perchlorate is important to the gas generant of the invention due to its gaseous decomposition and lack of particulate The potential problem of HCl generation production. may be overcome through the use of copper chromite and/or iron oxide as a catalyst and/or the sodium from the sodium nitrate. One aspect of the invention is the discovery that AP, which is a component that the industry has a propensity to avoid due to HCl generation, is useful in the inventive gas generants. As set forth in the Examples, the gas generants of the invention produce barely detectable levels of chloride containing gases.

The ratio of oxidizer to fuel in the gas generant is adjusted such that the amount of oxygen allowed in the equilibrium exhaust gases is from zero to 2 or 3% by volume, and more preferably from zero to 2.0% by volume.

The gas generant composition may optionally contain up to about 1.0 weight %, typically between about 0.1 and about 0.3 weight %, of iron oxide, copper chromite or mixtures thereof as catalysts. 5 Copper chromite (CuCr) has known properties as a catalyst. It is a mixed oxide of copper and chromium obtained by igniting copper ammonium chromate under controlled conditions. Barium is frequently added to prevent poisoning of the catalyst, however, the CuCr 10 used in the present invention is preferably free of barium. Copper chromite is principally used for the reduction of carboxyl groups (e.g., ketones to alcohols, and esters to alcohols). The preferred level of copper chromite in the inventive composition 15 is about 0.25 weight %. The iron oxide (Fe2O3) useful

The invention will now be described in greater detail by way of specific examples.

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oxide and CuCr may vary from about 1 to 10 microns.

the usual methods. The particle size of the iron

in the inventive compositions may be obtained by all

Referring to FIG. 1, there is represented in exploded view, an inflator 10 employed in testing several of the gas generant compositions disclosed herein. A first housing member 12 and a second housing member 22 are attached to one another through "friction or inertia welding". The inflator 10 also comprises an inventive strip filter 14, an enhancer tube 16, a squib with enhancer cup 18 and a room temperature vulcanizing rubber seal 20. A bed of gas generant pellets 30 is disposed between the strip filter 14 and the enhancer tube 16. Metal foil, not shown, lines the annular surface of the first

housing 12 covering gas exit portals 34 in the first housing.

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With reference to Figs. 2A and 2B, there is represented two embodiments of the inventive filter strip 14. Both embodiments of the filter strip contain at least three segments wherein the first segment 28 (a.k.a. the inside portion) has two rows of apertures therethrough positioned along each edge of the ribbon, an expanded metal segment 29 and second segment 15 wherein at least one row of apertures are present. FIG. 2A is an embodiment wherein the second segment 15 has two rows of a plurality of apertures 26 therethrough with diameter of about 2.0 mm. represents a second embodiment where the second segment 15 has a single row of apertures 26 therethrough with a diameter of about 4 mm. placement of the apertures is important for complete combustion of the generant. The first segment, which is adjacent the generant bed, requires apertures along each edges, while the second or final segment must have the aperture in the center of the ribbon. The size and number of the apertures can be varied to control the desired combustion level (i.e., rate of pressure generation). In use, the filter strip is coiled or rolled into a tubular configuration that is placed inside the inflator 10.

A metallic filter strip or ribbon with a combination of segments with holes and a segment of expanded metal can economically produce a filter that effectively cools the gas and removes particulates and slag generated when the gas generant is burned. The metal from which the filter strip 14 is produced can be any metal with a melting point high enough to

survive the combustion of the gas generant. The thickness of the strip can range from about 0.25 mm to 1.27 mm with about 0.51 mm to 0.76 mm being more preferred, about 0.63 mm being the most preferred.

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The length and height of the strip can vary widely depending upon the size and configuration of the inflator housing into which it is placed.

Dependent on the size of the housing, the filter strip is designed such that first segment 28 will complete the first turn during the formation of the coil and the expanded metal segment 29 will complete at least two turns of the coil. Preferably, the expanded metal segment 29 will complete at least three turns. The second segment 15 is of such length that it will completely circumferentially cover the outside of the coil.

Another important aspect of the filter strip is that apertures 24 in the first segment 28 are not aligned with, and do not overlay, the apertures 26 in the second segment 15. In the embodiment set forth in FIG. 2A, the apertures 24 are disposed towards the outside edge of segment 28 while the apertures 26 in the second segment 15 are disposed towards the interior. This aspect is important as it aids in creating a tortuous path for the gases. Further, the use of the expanded metal segment provides a large surface area for the capture of particulates and cooling of the gas and also creates a tortuous path for the gases.

As mentioned previously, the expanded metal segment 29 should be long enough to accomplish at least two turns during the formation of the coil. The diamond shaped openings in the expanded metal

segment 29 should have a dimension of about 0.04 to 0.12 mm by 0.32 to 0.8 mm. The expanded metal strip can be made by die cut stamping and the apertures can be drilled or stamped out.

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FIG. 3 is a cross section of an inflator housing taken along line 3-3 of FIG. 1 except that the squib with enhancer cup 18 is not shown in cross section. The bed of gas generant 36 is not shown for clarity. The inflator housing 10 comprises a first housing member 12 and a second housing member 22 that, in this representative embodiment, are attached by a spin weld 32. Other forms of attachment such as threaded engagement, laser welds and mechanical fasteners, are within the scope of the invention. The filtration strip 14 in coiled configuration, is shown as having five turns in FIG. 1. The apertures 24 through the first segment 28 can be in other arrangements than shown, i.e., in a random pattern, provided the apertures 24 are not directly across from the apertures 26 through the second segment. This is required so that the combustion gas must take a tortuous path through the expanded metal to the apertures 26 and then through the exit portals 34.

One additional aspect of the invention is that through subtle changes in the levels of the various components, the combustion temperature and igniting behavior of the generant can be modified to function in a variety of inflator configurations. As those skilled in the art will appreciate, changing the combustion level and temperature will change the CO and NOx content of the combustion gas as well as output. As an example, reduction of the combustion temperature by using a coolant, on the one hand, gives

disadvantages relating to CO and NOx content as well as output levels. On the other hand, at high output temperature, it leads to potential disadvantages with respect to damage to the airbag. Gas generant development should be understood to be a task of balancing contradicting properties in order to fulfill very special requirements.

In addition, it should be considered that reaction behavior of a gas generant, in areas other than basic chemistry, depends on igniting behavior, combustion surface area and design of the inflator housing which influences pressure build-up. Lastly, the design of the inflator housing can influence the properties of the gas generated through pressure build up as a result of filtering capabilities.

#### EXAMPLE I

#### Preparation of Gas Generant

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A one Kg batch of a gas generant composition was formulated according to Table I below. The compositions were prepared by grinding the individual components (when needed, i.e., NaN) to a particle size of less than 100 microns and then all of the components of the generant were sifted and then blended in a Turbula® mixer (manufactured by W.A.B. of Switzerland). Mixing continued for one (1) hour. The material was then pelletized with a rotary pellet press. The pellets were about 5 mm in diameter, 1.8 mm high, weighed about 55 to 65 mg each and had a density of about 1.6 to 1.7 g/cm3.

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The formed pellets were then loaded into steel inflators of the type shown in FIG. 1. Either about 19 or 23 gms of the pellets were loaded into each of the steel housings. The 19 gm charge of generant was for a 40 liter airbag while the 23 gm charge was for a 60 liter airbag. The burst foil or tape comprises a thin sheet (about 0.005 mm. thick) of stainless steel with an adhesive on one side. The adhesive side of the burst foil is placed against the inside surface of the inflator housing so as to hermetically seal all of the apertures 34. The apertures 34 are exhaust ports for the gases generated by the generant and were about 2.4 mm in diameter for the 40 liter airbag and about 2.5 mm for the 60 liter airbag. The number of apertures 34 was four. The test inflator housing had a total volume of about 88 cm<sup>3</sup>, while the region of the housing located inwardly of the filter and containing the pellets of gas generating material had a volume of about 46 cm<sup>3</sup> for the 40 liter airbag and about 46 cm<sup>3</sup> for the 60 liter airbag. The inflator also incorporated about 0.9 g of  $BKNO_3$  (a mixture of boron nitrate and potassium nitrate, conventionally used in the industry), as an enhancer and was associated with the squib with enhancer cup 18.

#### EXAMPLE II

#### Testing of the Gas Generant

5 Two assembled inflators containing 19 gms of the inventive gas generant pellets (Sample No. 5) were evaluated in a 2.8 cubic meter test chamber fitted with equipment to record the pressure and time profile of the combustion and to analyze the gases exiting the 10 inflator. The amount of particulate or slag produced by the burning generant was also determined using standardized techniques. The inflators were installed into the test chamber and the gas generant pellets were ignited. The temperature of the inflator at 15 firing was about 23°C + 2°C at a relative humidity of about 43%. Immediately after firing of the inflator, gas samples were withdrawn from the test chamber for analysis by FTIR (Fourier Transform Infrared Spectroscopy).

Airborne particulate production was measured by filtering post ignition air from the test chamber through a fine filter and measuring the weight gained by the filter. The average total airborne particulate mass for the two tests was 6.85 mg. The average total particulate concentration for the two tests was 68.5 mg/m<sup>3</sup>.

#### Gaseous Reaction Products

The test chamber was attached to a vacuum pump, a bubble flow meter, filters and a FT/IR gas analyzer (spectrophotometer). Gas samples were analyzed using an FTIR spectrometer at zero time (before deployment)

and at 1, 5, 10, 15 and 20 minute intervals after ignition or via gas chromatography.

The ammonia, benzene, carbon dioxide, formaldehyde, hydrogen chloride, hydrogen cyanide, methane, sulfur dioxide, carbon monoxide (CO), nitric oxide (NO) and nitrogen dioxide (NO2) and water vapor levels of the gases produced in the 100 cubic foot test chamber for the two test samples are set forth in Table II. Samples were transferred directly to the FTIR gas cell from the 2.8 cubic meter test chamber via 2 meters of .6 cm OD fluoropolymer tubing.

The results set forth in Table II demonstrate that the gas generants of the present invention produce an acceptable gas for use in vehicle occupant restraint systems. The gas generants of the present invention produce a reasonably clean combustion gas and the pellets of the generant also resist degradation due to moisture and thermal cycling.

Both firings of the inflator demonstrated acceptable bag inflation, peak bag pressure and sustained bag pressure and thus would be useful in a vehicle airbag occupant safety system.

#### EXAMPLE III

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#### Thermal Stability

To test the thermal stability of the gas generant according to this invention, 1.0 gm of the Sample No. 5 composition from Table I was placed in a petridish and then in an oven at 135°C for two hours. The sample was removed and allowed to cool at room temperature. Inspection of the pellets revealed that

no melting of the gas generant composition had occurred and that the pellets were intact and did not evidence any cracking, crumbling or change in shape.

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#### EXAMPLE IV

#### Thermal Stability

In this experiment, 19 gms of Sample No. 5 was

placed in an inflator as set forth in Example II.

After assembly of the inflator, the unit was placed in
an oven at 107°C for two hours. The inflator was
removed from the oven, allowed to cool to room
temperature and then fired. The inflator performed

similar to the tests set forth in Example II, thus
demonstrating the thermal stability of the
compositions according to the invention.

#### EXAMPLE V

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#### Hot Cold Ignition

In this experiment, the ignition characteristics of the gas generant at 90°C, ambient (about 24°C) and -40°C was investigated. 19 gms of the generant Sample No. 5 was loaded into the housings. A total of nine inflators were prepared. Three were placed in an oven at 90°C for two hours and three were placed in a freezer at -40°C for two hours. Three inflators remained at room temperature. The inflators were fired at their respective soak temperatures in a 60 liter test chamber fitted to measure combustion gases, pressure and particulates. Plots of pressure

versus time were recorded. Table III sets forth the maximum chamber pressure, time to maximum pressure and area under the curve for each test.

The data evidence that the gas generant according to the invention provides satisfactory combustion properties over a wide range of temperatures to properly inflate the airbag.

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Total particulate production from each test was also collected. Following venting of the tank to the atmosphere, the interior of the 60 liter test chamber was carefully scrubbed and rinsed with deionized water to measure particulate production. The particulate produced by gas generants comprises a mixture of water soluble and insoluble reaction products. The aqueous mixture of the soluble reaction products and the insoluble dust were analyzed to determine total particulate production. Table IV sets forth the insoluble, soluble and total particulates for each run.

The data evidence that the gas generant composition used in an inflator according to the invention produces a relatively clean gas upon combustion; that is, from a 19 gm charge of generant, less than 1.5 gms of solids exit the inflator.

Toxicity testing was also conducted on the ambient firings of the generant and the results are set forth in Table V.

These data indicate that the generant according to the invention produces a gas that is relatively non-toxic and would therefore be useful in the inflation of air bags and as fire extinguishers.

From these experiments and others that are being conducted at the time of the filing of this

application, it is clear that the gas generant according to the invention is useful for inflating airbags and can also be used as fire extinguishers. The generants of the invention are virtually unaffected by temperature extremes and possess excellent ignition and combustion properties.

Surprisingly, the use of ammonium perchlorate (AP) does not cause a chlorine problem in the combustion gas. This is quite an unexpected result to those skilled in the art.

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#### CLAIMS:

- 1. An inflator (10):
- (a) a housing means defining at least one
  5 discharge port (34) for directing a gas flow from said housing means (12, 22);
  - (b) a gas generating (30) means disposed within said housing means;
    - (c) an ignition unit; and
- 10 (d) a filter means (14) between said discharge port and said gas generating means; said filter means comprising a metallic ribbon with at least one segment of its length having apertures (24) therethrough and at least one segment of its length comprising expanded metal configured into a coil.
  - 2. The inflator (10) according to claim 1 wherein said gas generating means (30) is a gas generant composition comprising: a fuel component which is used at a level between 45 and 70 weight %, which comprises a mixture of at least two fuels selected from the group consisting of guanidine nitrate (GN), nitroguanidine (NG), triaminoguanidine nitrate (TAGN), diaminoguanidine nitrate (DAGN) and monoguanidine nitrate (MGN); and an oxidizer component which is used at a level of between 25 and 50 weight %, which comprises a mixture of alkali metal nitrates, alkaline earth metal nitrates, and ammonium perchlorate.

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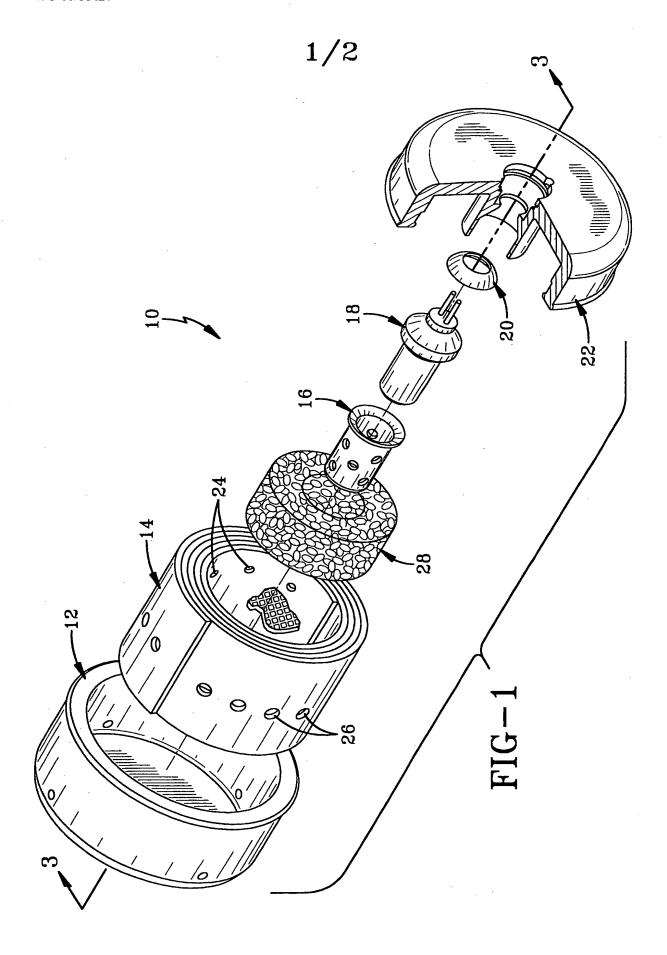
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3. The inflator (10) according to claim 2 wherein said catalyst is selected from copper chromite, iron oxide and mixtures thereof.

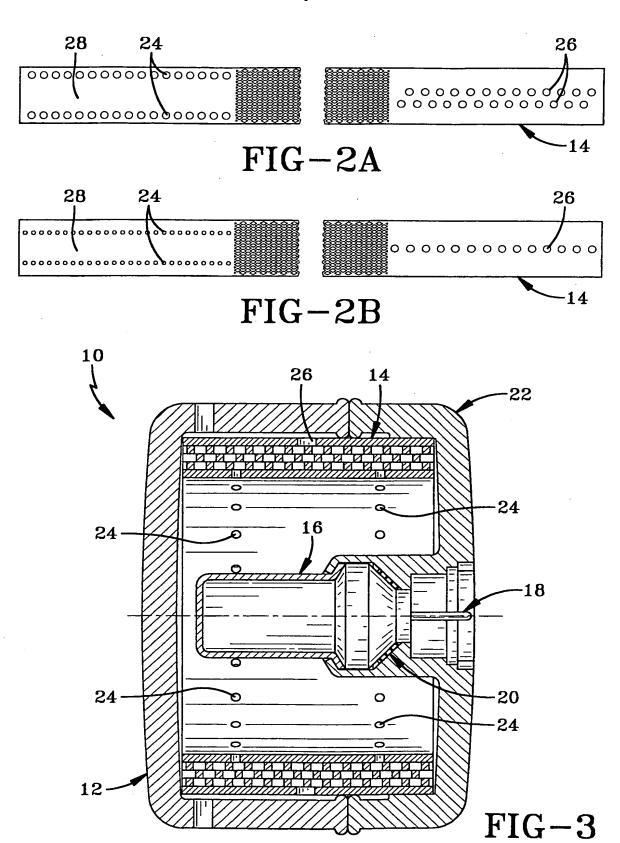
4. The inflator (10) according to claim 3 wherein: said fuel component comprises a mixture of NG and GN; said oxidizer component comprises a mixture of strontium nitrate (SN), ammonium perchlorate (AP) and sodium nitrate (NaN); and said catalyst is copper chromite (CuCr).

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- 5. The inflator (10) according to claim 4
  wherein: said NG is at a concentration of
  10-20 weight % and said GN is at a concentration of
  35-50 weight %; said SN is at a concentration of
  5-15 weight %; said AP is at a concentration of
  15-25 weight %; said NaN is at a concentration of
  5-25 weight %; andsaid copper chromite is at a
  concentration of 0.1-0.5 weight %.
- 6. The inflator (10) according to claim 5
  wherein: said NG is at a concentration of
  14-17 weight % and said GN is at a concentration of
  40-43 weight %; said SN is at a concentration of
  7-10 weight %; said AP is at a concentration of
  21-24 weight %; and said NaN is at a concentration of
  10-13 weight %; and said CuCr is at a concentration of
  0.2-0.3 weight %.
- 7. The inflator (10) according to claim 1
  wherein said metallic ribbon comprises a first segment
  30 having apertures (24)therethrough at the edges of said
  ribbon, and a second segment having apertures
  (24)therethrough wherein said apertures are centrally
  located.



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# INTERNATIONAL SEARCH REPORT

----0 (second sheet)(July 1992)\*

International application No. PCT/US99/14943

	SIFICATION OF SUBJECT MATTER		
HS CI .	360R 21/26; C06B 31/12 280/741, 149/36, 62, 76		
According to	International Patent Classification (IPC) or to both as	ational classification and IPC	
	DS SEARCHED		
Minimum do	cumentation searched (classification system followed	by classification symbols)	
	280/741, 149/36, 62, 76		
Documentati	on searched other than minimum documentation to the e	extent that such documents are included	in the fields searched
	ata base consulted during the international search (nan anded metal, 280/clas	ne of data base and, where practicable,	search terms used)
C. DOC	UMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where app	ropriate, of the relevant passages	Relevant to claim No.
Α	US 3,904,221 A (SHIKI et al.) 09 Sept	tember 1975 (09/09/75)	1
A	US 3,958,949 A (PLANTIF et al.) 25	1	
Α	US 4,858,951 A (LENZEN) 22 August	1	
Α	US 5,104,466 A (ALLARD et al.) 14	1	
A	US 5,443,286 A (CUNNINGHAM (22/08/95)	1	
A	US 5,503,806 A (FULMER et al.) 02	April 1996 (02/04/96)	1
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#### INTERNATIONAL SEARCH REPORT

International application No. PCT/US99/14943

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.	
A	US 5,551,724 A (ARMSTRONG III et al.) 03 September 1996 (03/09/96)	1.	
Α	US 5,609,360 A (FAIGLE et al.) 11 March 1997 (11/03/97)	1 .	
A	US 5,665,131 A (HOCK et al.) 09 September 1997 (09/09/97)	1	
A	US 5,739,460 A (KNOWLTON et al.) 14 April 1998 (14/04/98)	2	
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P, A	US 5,765,866 A (CANTERBERRY et al.) 16 June 1998 (16/06/98)	1	
P, A	US 5,773,754 A (YAMATO) 30 June 1998 (30/06/98)	2	
P, A	US 5,806,888 A (ADAMINI) 15 September 1998 (15/09/98)	1	
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